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Association of metals with plastic production pellets in the marine environment

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ABSTRACT

Plastic production pellets sampled from four beaches along a stretch of coastline (south Devon, SW England) and accompanying, loosely adhered and entrapped material removed ultrasonically have been analysed for major metals (Al, Fe, Mn) and trace metals (Cu, Zn, Pb, Ag, Cd, Co, Cr, Mo, Sb, Sn, U) following acid digestion. In most cases, metal concentrations in composite pellet samples from each site were less than but within an order of magnitude of corresponding concentrations in the pooled extraneous materials. However, normalisation of data with respect to Al revealed enrichment of Cd and Pb in plastic pellets at two sites. These observations are not wholly due to the association of pellets with fine material that is resistant to ultrasonication since new polyethylene pellets suspended in a harbour for 8 weeks accumulated metals from sea water through adsorption and precipitation. The environmental implications and potential applications of these findings are discussed.

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1. Introduction

Although plastics have been recognised as an important component of marine litter for several decades, their ecological and biological impacts have only been acknowledged and understood recently (Derraik, 2002; Moore, 2008). Pre-production pellets, or resin pellets, the raw materials from which plastics are moulded, are a common, global form of litter encountered on the strandline of beaches and in suspension in coastal and oceanic waters (Gregory, 1983; Shiber, 1987; Khordagui and Abuhilal, 1994; McDermid and McMullen, 2004). Pellets are lost during loading and transportation, both on land and at sea, and during their handling at plastic moulding factories (Redford et al., 1997). Due to their buoyancy and durability, lost pellets may be transported considerable distances in the oceans before becoming temporarily or permanently stranded (Ivar do Sul et al., 2009).

Descriptions of beached litter commonly report heterogeneous assortments of plastic production pellets (Endo et al., 2005; Rios et al., 2007). They are generally 2–5 mm in diameter and are most commonly polyethylene or polypropylene, are usually disc-, ovoid- or cylindrical-shaped and may be colourless, translucent or coloured (Endo et al., 2005; Rios et al., 2007). Aging of pellets is usually accompanied by discolouration (e.g. yellowing), abrasion, cracking, fouling, tarring and encrustation by precipitates (Endo et al., 2005).

Compared with larger forms of litter, plastic production pellets are more difficult to clear from a beach but are aesthetically less obtrusive. The main ecological risk associated with pellets, how-

ever, appears to be their inadvertent (or sometimes selective) ingestion by animals, including birds, fish and invertebrates, resulting in diminished foraging ability and feeding stimulus, loss of nutrition and intestinal blockage (Ryan, 1987; Toda et al., 1994; Provencher et al., 2009; Graham and Thompson, 2009). Pellets are also carriers of organic contaminants, including components of the plastic itself (e.g. plasticisers) and persistent, hydrophobic compounds, such as polychlorinated biphenyls and polycyclic aromatic hydrocarbons, that sorb onto or into the pellets during their transport in the aqueous phase (Mato et al., 2001; Endo et al., 2005; Karapanagioti and Klontza, 2008).

In the present study, we examine the association and interactions of new and beached production pellets with a variety of metals. Although pure polymers are generally acknowledged to be rather inert towards aqueous cations, loss of trace metals to container surfaces during the storage of water samples is a common problem (Giusti et al., 1994; Cobelo-García et al., 2007). Moreover, adsorption of metals to polymers is predicted to be enhanced as the material ages and its polarity, surface area and porosity increase and it becomes fouled with organic matter and hydrous metal oxides. Thus, it is possible that plastics may serve as a means of metal transport in the marine environment, and that metal signatures of plastics may afford a means of tracing the provenance and age of the litter.

2. Materials and methods

2.1. Sample sites

Twelve beaches along a 40 km stretch of coastline between Plymouth and the Kingsbridge estuary (Devon, SW England) were

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visited between May and October, 2009. Pellets were subsequently sampled from four beaches, shown in Fig. 1 and described below, selected on the basis of contrasting size, geomorphology, orientation, uses, and variety and abundance of pellets. Soar Mill Cove is a small, remote sandy inlet that faces southwest. It is owned (and protected) by the National Trust and is popular with bathers during the summer. Thurlestone is a wide, expansive sandy beach facing southwest that is also owned by the National Trust and is backed by a nature reserve. It is a popular destination for non-motorised water sports. Bovisand is a sheltered, gently-sloping sandy bay facing west towards Plymouth Sound that serves the residents of Plymouth (250 000) throughout the year. Saltram is an intertidal silt-sandflat on the east bank of the Plym estuary. The estuary is urbanised along most of its 5 km tidal axis and receives treated sewage effluent and landfill leachate during ebb flow.

2.2. Sampling

Several hundred pellets were retrieved from the high tide line and berm zone of each beach using plastic tweezers and were stored as composite samples in acid-cleaned 50 ml screw-capped polypropylene centrifuge tubes. In the laboratory, 100 pellets from each site (or 4×100 pellets from Soar Mill Cove for replication purposes) were transferred to clean centrifuge tubes before 25 ml of English Channel sea water, collected in bulk and available on tap in the laboratory following on-line filtration through $0.6 \mu\text{m}$, was added. The contents were ultrasonicated for 5 min to remove extraneous material (loosely adhered or entrapped debris) before being sieved through a 1 mm nylon mesh. Material passing the sieve was filtered through $0.45 \mu\text{m}$ using an acid-cleaned polyethylene Buchner filtration unit connected to a vacuum pump. Filters and pellets were then air-dried in Perspex Petri dishes at 45°C for 48 h.

2.3. Suspension experiment

In order to examine the physical and chemical changes conferred on plastics in sea water, new polyethylene pellets (white, diameter $\sim 4 \text{ mm}$, mass $\sim 25 \text{ mg}$), sourced from a local moulding factory (Algram Group Ltd, Plymouth), were suspended in a Sutton Harbour, Plymouth (Fig. 1) for a period of 8 weeks (October–December, 2009). This was achieved by tying a nylon-mesh bag ($15 \text{ cm} \times 10 \text{ cm}$) containing 100 pellets to a mooring chain just below the water surface. Pellets retrieved from the bag were air-dried as above, and divided into four subsamples.

2.4. Pellet analysis

The polymers of 30 individual beached pellets of various colours and shapes were identified by Fourier transform-infrared (FTIR) spectroscopy using a Bruker IFS 66 spectrometer attached to a Hyperion 1000 IR microscope with a liquid nitrogen cooled mercury–cadmium–telluride detector. Slices of each pellet were cut using a stainless steel scalpel and then compressed between the windows of a Specac diamond compression cell until an appropriate thickness was attained. Transmission spectra were acquired by averaging 100 scans at a resolution of 4 cm^{-1} over the range $4000\text{--}400 \text{ cm}^{-1}$. A qualitative evaluation of the age of the pellets was ascertained from the carbonyl index; that is, the height of the carbonyl peak measured at 1717 cm^{-1} relative to the height of a reference peak (unaffected by weathering) measured at 1465 cm^{-1} (Artham et al., 2009).

The surface characteristics of selected pellets were examined by scanning electron microscopy–energy dispersive X-ray spectroscopy (SEM–EDS). Pellets were sputter-coated with a thin film in an EMITECH K 450X high vacuum carbon-coating unit before being attached to the SEM with adhesive tape. Samples were photographed using a JEOL JSM-6100 operated at 20 kV and at a working

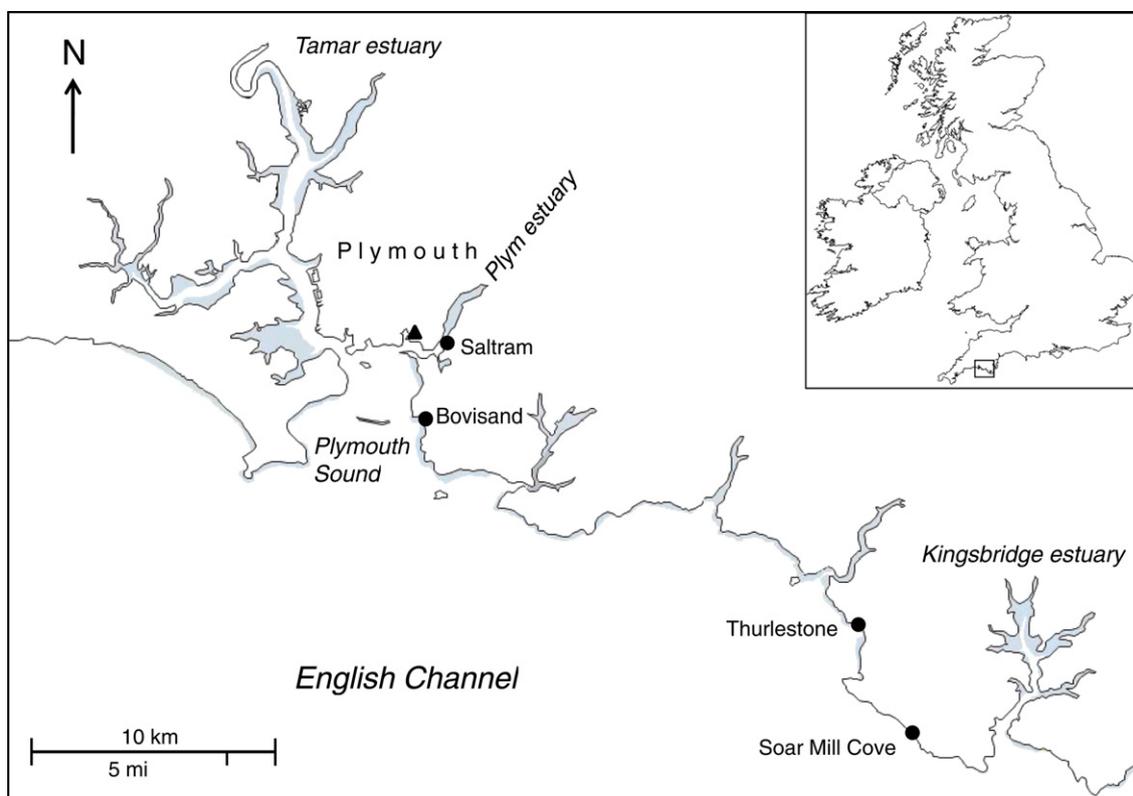


Fig. 1. The beaches and intertidal areas of south Devon, SW England (shaded), and the locations at which pellets were sampled (●) and where the suspension experiment was conducted (▲).

distance of 15 mm. Qualitative elemental analysis was performed using an Oxford Instruments Inca 200 system.

2.5. Sample digestion

One hundred pellets from each beach that had been ultrasonicated in sea water, 100 new pellets, three samples of 25 pellets each resulting from the suspension experiment and filters containing extraneous material retrieved by ultrasonication were weighed into individual polypropylene centrifuge tubes. Twenty millilitres of a mixture of three parts 2 M HCl to one part 3 M HNO₃ (both VWR AnalaR) were added to each sample contained in a clean centrifuge tube. The screw-capped contents were then agitated laterally at about 100 rpm overnight and at room temperature before digests were decanted into clean tubes pending analysis. In order to assess the accuracy of subsequent metal analysis (see below), triplicate 250 mg portions of reference sediment (LKSD 4; Canadian Certified Reference Materials Project) that was certified for metal concentrations available to aqua regia were digested in 7 ml of the undiluted acid mixture for 2 h in covered, acid-cleaned 30 ml Pyrex beakers at 75 °C on a hotplate. These digests were transferred to 25 ml volumetric flasks and diluted to mark with Millipore Milli-Q water.

2.6. Digest analysis

Digests were analysed for Al, Fe, Mn, Cu, Cr, Pb and Zn by inductively coupled plasma-optical emission spectrometry (ICP-OES) using a Varian 725 ES (Mulgrave, Australia), and for Ag, Cd, Co, Mo, Sn, Sb and U by inductively coupled plasma-mass spectrometry (ICP-MS) using a Thermo Scientific X Series II bench top mass spectrometer (Thermo Elemental, Winsford, UK). Both instruments were calibrated using mixed, acidified standards, and internal standardisation was achieved by the addition of yttrium (ICP-OES) and indium and iridium (ICP-MS). Analysis of the aqua regia digests of the reference sediment revealed metal concentrations that were within 10% of certified values with the exception of Al, Fe, Co and Mo. Extraction of between 70 and 80% of the latter metals presumably reflects their more refractory nature in the sediment matrix.

3. Results

3.1. Distribution and physical characteristics of pellets

Plastic production pellets were visible on all of the beaches visited with the exception of a sheltered, south-facing cove, were most abundant around the strand line and berm (exceeding 100 per m² in many cases) and were nearly always the dominant form of visible litter or plastic on a number basis. Pellets could be categorised as flat ovoids–spheruloids or ribbed (or concertinaed) cylinders. Extraneous matter was most evident within the grooves of the ribbed variety and the quantity retrieved by ultrasonication of the composites totalled about 100 mg in each case, or about 1 mg per pellet on average. Pellets were generally 3–5 mm in diameter or height and displayed a variety of colours; we noted a greater proportion of white/off white and bright yellow pellets on the eastern beaches and an increasing proportion of black, grey, blue and green pellets towards the west. With respect to the beaches sampled, average pellet mass after ultrasonication (± 1 standard deviation) was 36.8 ± 11.9 mg at Soar Mill Cove, 39.2 ± 25.2 mg at Thurlstone, 33.1 ± 7.5 mg at Bovisand and 32.0 ± 8.2 mg at Saltram.

The precise origins of the pellets are unclear but multiple and varied sources are likely. Given the persistence of pellets in the marine environment (estimates vary from a few years to several

decades; Derraik, 2002), many are likely to have distant origins, being transported to the SW of England via ocean currents. The region itself is a relatively small producer of plastics and has a relatively low plastics expertise (Mortimore et al., 2008). Nevertheless, a handful of primary plastic manufacturers are located to the east of the area surveyed and several moulding plants are located in Plymouth; clearly, therefore, pellets spilled during handling, transportation or processing have the propensity to enter the local marine environment via stormwater runoff. A more specific source of pellets to the region is the MSC Napoli, a cargo ship that ran aground in Lyme Bay, about 80 km to the NE of the region, in January 2007 (Guitart et al., 2008). According to local newspaper reports, amongst cargo that was lost were several bags of yellow plastic pellets. A westward reduction in the abundance of bright yellow pellets on the beaches sampled is consistent with the nature and location of this source.

FTIR analysis of 30 pellets of different colour and shape identified polyethylene as the principal plastic ($n = 29$; the remaining pellet being nylon) and significant variations in the carbonyl index, hence degree of aging. Aging was accompanied by yellowing or greying of clear, translucent and white pellets, and SEM analysis revealed that aged pellets displayed various degrees of cracking, fissuring and chalking (effects absent in new pellets), had surface precipitates of both chemical and biological origin, but exhibited no tarring. SEM images and accompanying EDS spectra of a new pellet and an ovoid-shaped pellet sampled from Thurlstone are compared in Figs. 2a and 2b.

3.2. Metal concentrations of pellet composites

Concentrations of major metals (Al, Fe and Mn) and trace metals (including the trace metalloid, Sb) available to dilute mineral acid digestion in the composite pellet samples and in extraneous (adherent and entrapped) solids from each location are shown on a dry w/w basis in Table 1. Regarding the replicates from Soar Mill Cove that were digested independently, relative standard deviations range from less than 10% (Al, Fe, Sn) to about 50% (Cu, Zn, Cd) in the pellets and from less than 5% (U) to 40–50% (Cd, Sb) in the extraneous solids. Metal concentrations are nearly always greater in the extraneous solids than in the pellets because components of the former, principally silt grains and algal fragments, have greater surface areas, porosities and densities of charged surface sites for the accommodation of metals; moreover, acid-soluble metal-bearing minerals are an intrinsic component of sediment particles. What is perhaps surprising, however, is that, for several metals, concentrations in the pellet composites are within an order of magnitude of the concentrations in the extraneous solids, and in the case of Pb at Saltram, the concentration in the pellets exceeds that in the extraneous solids.

For most metals, concentrations in the pellet composites also increase westward, an effect that is not as pronounced or is not evident for metals in the extraneous solids. This gradient is associated with both an increasing proximity to anthropogenic sources of metals in the Plymouth region of the south Devon coast and an increasing proportion of coloured pellets (mainly black, green and blue, but not bright yellow) among the composite samples.

3.3. Enrichment factors for trace metals in pellets

The origins of the extraneous solids associated with the plastic production pellets are not clear, but are likely to be local (debris from beaches) and non-local (precipitates and fine suspended particles accumulated during transport in sea water). Assuming that extraneous matter affords an indication of environmental exposure of pellets to particulate metals, enrichment factors, EF, were computed for each site as follows:

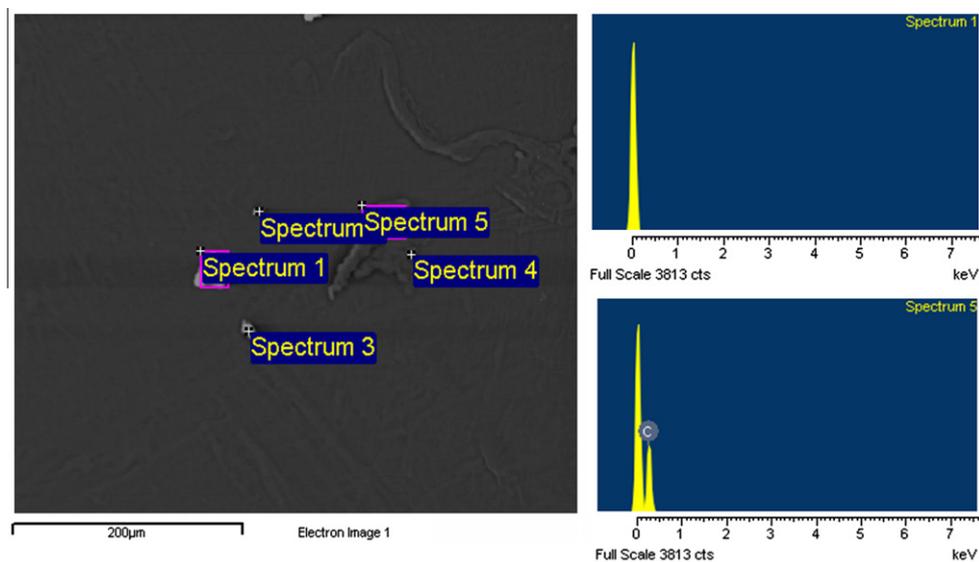


Fig. 2a. SEM image and selected, accompanying EDS spectra of a new polyethylene pellet.

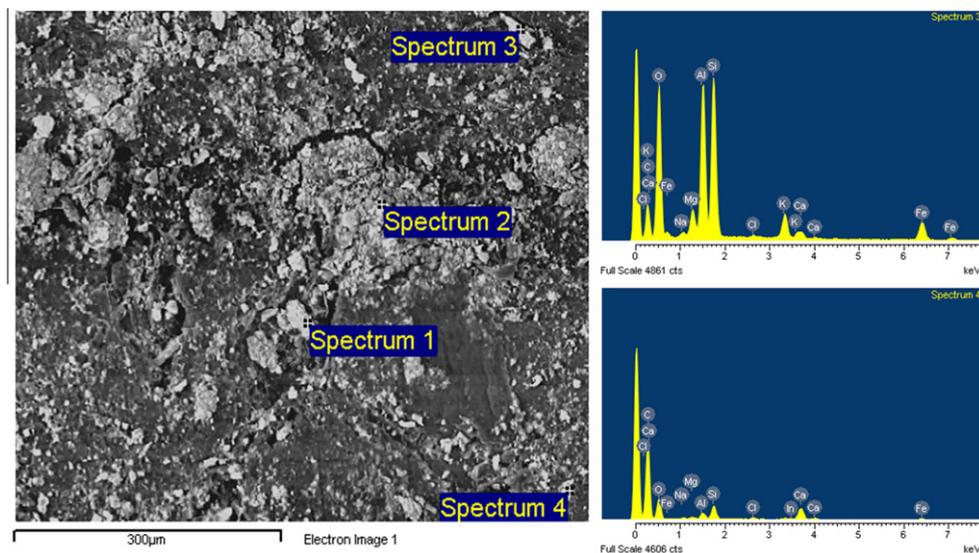


Fig. 2b. SEM image and selected, accompanying EDS spectra of an ovoid-shaped, off-white polyethylene pellet sampled from Thurlestone.

Table 1

Concentrations of metals in composite pellet samples and in extraneous solids from the different sampling locations that were available to acid digestion. Errors for the Soar Mill sample represent the standard deviation arising from four replicate digestions.

	Pellets				Extraneous solids			
	Soar Mill	Thurlestone	Bovisand	Saltram	Soar Mill	Thurlestone	Bovisand	Saltram
Al, $\mu\text{g g}^{-1}$	7.05 ± 0.66	9.44	17.61	49.79	44.56 ± 5.73	17.99	70.17	132.87
Fe, $\mu\text{g g}^{-1}$	25.85 ± 2.09	35.67	54.60	64.97	203.69 ± 48.43	101.64	199.67	324.93
Mn, $\mu\text{g g}^{-1}$	1.58 ± 0.57	1.28	2.01	8.31	19.38 ± 6.12	5.57	13.00	23.54
Cu, $\mu\text{g g}^{-1}$	0.06 ± 0.03	0.14	0.29	0.61	4.60 ± 0.60	4.57	6.33	5.85
Pb, $\mu\text{g g}^{-1}$	0.15 ± 0.04	0.41	0.73	1.08	0.74 ^a	0.86	1.33	0.28
Zn, $\mu\text{g g}^{-1}$	0.55 ± 0.27	0.42	0.94	2.34	6.67 ± 1.09	4.57	6.50	10.31
Ag, ng g^{-1}	2.4 ± 0.4	4.9	6.0	30.0	484 ^a	675	298	266
Co, ng g^{-1}	27 ± 5	25	53	101	331 ± 102	78	197	294
Cd, ng g^{-1}	1.7 ± 0.8	10.0	3.6	5.0	52.0 ± 24.9	7.4	6.8	21.0
Cr, ng g^{-1}	19 ± 5	63	69	151	598 ± 50	571	667	836
Mo, ng g^{-1}	7 ± 2	7	11	15	427 ± 41	295	318	352
Sb, ng g^{-1}	7 ± 1	6	14	17	88 ± 36	34	123	89
Sn, ng g^{-1}	18 ± 1	28	46	114	365 ± 30	342	462	680
U, ng g^{-1}	4.5 ± 2.1	2.8	4.0	10.0	170 ± 7.0	97	108	124

^a Ag and Pb were only detected in one digest of extraneous solids from Soar Mill.

$$EF = ([Me]_p/[Al]_p)/([Me]_x/[Al]_x) \quad (1)$$

Here, $[Me]_p$ and $[Me]_x$ represent the w/w concentrations of a metal in the composite pellet sample and in extraneous matter, respectively, and Al is used to normalise for particle grain size effects or the extent of Al coverage (by, for example, aluminosilicates) of the plastic. Values of EF shown in Table 2 indicate, for some metals, a westward increase that is not as clear as that ascertained from the corresponding absolute metal concentrations and, at each site, a range among the different metals that spans nearly two orders of magnitude. Cadmium and Pb exhibit enrichment in pellets relative to extraneous solids in two samples each; in all remaining cases, however, EF is less than unity.

3.4. Metal accumulation by suspended pellets

New polyethylene pellets that had been suspended in harbour sea water for a period of 8 weeks were partly discoloured and speckled in appearance due to the presence of organic and inorganic precipitates on the surface of the plastic. Concentrations of metals that were detectable in acid digests of these pellets, after correction for metal concentrations encountered in equivalent digests of clean pellets, are shown in Table 3. For a given metal, concentrations are variable among replicates, presumably reflecting the heterogeneity of the accumulation process(es). The order of metal accumulation ($Fe > Al > Mn > Pb > Cu, Zn > Ag$) is similar to the order of metal concentrations in the beached pellet composite samples, and mean concentrations are within an order of magnitude of the corresponding concentrations in pellets sampled from

Saltram (the site closest to the location of the suspension experiment; Fig. 1).

4. Discussion

Despite the general conception that plastics are relatively inert towards aqueous metals, this study has shown that production pellets are able to accumulate metals to concentrations that, in some cases, approach those on extraneous solids (principally sediment and algal fragments). This observation is not consistent with compositional, morphological or surface area considerations. Thus, sediment is composed of charged minerals of relatively high surface area (several $m^2 g^{-1}$ according to BET nitrogen adsorption; Turner et al., 2001) that readily accommodate metals and release them in dilute mineral acids, while the surface of production pellets is hydrophobic and its area is typically only a few tens of $cm^2 g^{-1}$ when new (Mato et al., 2001). Deviation of Al-normalised trace metal enrichment factors from unit value implies that metal accumulation by the pellets is not simply the result of adherence or entrapment of local solid material (at least when beached) that is resistant to ultrasonication. Results of suspension experiments clearly indicate the propensity of pellets to accumulate metals directly from the water column or surface microlayer. Possible mechanisms of metal uptake include direct adsorption of cations or complexes onto charged sites or neutral regions of the surface of the plastic, an effect that is commonly observed during the storage or processing of water samples (Giusti et al., 1994; Fischer et al., 2007; Cobelo-Garcia et al., 2007), and co-precipitation with or adsorption onto hydrous oxides of Fe and Mn. Since the order of metal accumulation or enrichment does not follow that of hydrolysis constants, affinities for metal oxides in sea water or the abundance of metals in ferromanganese oxide deposits in the oceans (Chester, 1990), however, mechanisms are likely to be varied and complex.

The importance of pellets as reservoirs for metals is relatively low on beaches and estuarine sand- or siltflats, even those containing the highest densities of plastics. However, given their size and buoyancy, pellets afford a means of ready transportation of metals and other contaminants to open surface waters where their relative importance in suspension is much greater (Moore et al., 2002). Moreover, given that metals on pellets are adsorbed to the surface of plastic or associated with hydrogeneous or biogenic phases, they are likely to occur in a relatively bioaccessible form to fauna that inadvertently ingest them. The results of this study also raise the distinct possibility that other micro- and macroplastics accumulate and transport metals in the marine environment and may serve as indicators of metal contamination. Conversely, it is also conceivable that the metallic content of adsorbed and precipitated metals may serve as tracers for the age and origin of plastics in the marine environment. Clearly, further investigations are required into the mechanisms and kinetics of plastic-metal interactions and the effects of different environmental variables on these processes.

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Table 2

Trace metal enrichment factors, calculated using Eq. (1), for the different sampling locations. Errors for the Soar Mill sample represent the standard deviation arising from four replicate sample digestions.

Location	Pellets			
	Soar Mill	Thurlestone	Bovisand	Saltram
Cu	0.21 ± 0.08	0.06	0.18	0.28
Pb	0.14 ^a	0.92	2.17	10.4
Zn	0.54 ± 0.26	0.18	0.57	0.61
Ag	0.02 ^a	0.01	0.08	0.30
Co	0.56 ± 0.21	0.61	1.07	0.92
Cd	0.24 ± 0.12	2.58	2.11	0.64
Cr	0.21 ± 0.08	0.21	0.41	0.48
Mo	0.11 ± 0.06	0.05	0.14	0.11
Sb	0.39 ± 0.06	0.34	0.45	0.51
Sn	0.31 ± 0.07	0.16	0.40	0.45
U	0.16 ± 0.06	0.06	0.15	0.22

^a Ag and Pb were only detected in one digest of extraneous solids from Soar Mill.

Table 3

Metal concentrations on new plastic (polyethylene) pellets that were suspended in harbour sea water for a period of 8 weeks. Errors represent the standard deviation arising from three replicate sample digestions.

Metal	Concentration
Al, $\mu g g^{-1}$	6.20 ± 3.65
Fe, $\mu g g^{-1}$	17.98 ± 7.49
Mn, $\mu g g^{-1}$	2.61 ± 1.97
Cu, $\mu g g^{-1}$	0.28 ± 0.18
Pb, $\mu g g^{-1}$	1.72 ± 0.92
Zn, $\mu g g^{-1}$	0.25 ± 0.18
Ag, $ng g^{-1}$	24.7 ± 9.4

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